

10/528179

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NEWS 15 DEC 18 MARPAT to CA/Caplus accession number crossover limit increased
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NEWS 17 DEC 27 CA/Caplus enhanced with more pre-1907 records
NEWS 18 JAN 08 CHEMLIST enhanced with New Zealand Inventory of Chemicals
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NEWS 21 JAN 16 WPIDS/WPINDEX/WPIX enhanced with IPC 8 reclassification data
NEWS 22 JAN 22 CA/Caplus updated with revised CAS roles
NEWS 23 JAN 22 CA/Caplus enhanced with patent applications from India
NEWS 24 JAN 29 PHAR reloaded with new search and display fields
NEWS 25 JAN 29 CAS Registry Number crossover limit increased to 300,000 in
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NEWS EXPRESS NOVEMBER 10 CURRENT WINDOWS VERSION IS V8.01c, CURRENT
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AND CURRENT DISCOVER FILE IS DATED 25 SEPTEMBER 2006.

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FILE LAST UPDATED: 25 Jan 2007 (20070125/ED)

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This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s silica gel

515517 SILICA

488363 GEL

L1 89869 SILICA GEL

(SILICA(W)GEL)

=> s l1 and liquid chromatograph?

682656 LIQUID

405792 CHROMATOGRAPH?

87558 LIQUID CHROMATOGRAPH?

(LIQUID(W)CHROMATOGRAPH?)

L2 4609 L1 AND LIQUID CHROMATOGRAPH?

=> s uncoated and l2

15952 UNCOATED

L3 2 UNCOATED AND L2

=> d ibib abs kwic

L3 ANSWER 1 OF 2 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 90:199722 CA

TITLE: Improvements in liquid chromatography column life and method flexibility by saturating the mobile phase with silica

AUTHOR(S): Atwood, J. G.; Schmidt, G. J.; Slavin, W.

CORPORATE SOURCE: Perkin-Elmer Corp., Norwalk, CT, USA

SOURCE: Journal of Chromatography (1979), 171, 109-15
CODEN: JOCRAM; ISSN: 0021-9673

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Liquid chromatog. column life was prolonged by equilibrating the mobile phase with silica using an appropriate column mounted in the oven ahead of the anal. column. An atomic absorption method was adapted for the detection of Si eluting from the column to measure quant. the loss of silica for different anal. conditions. An injection procedure which permitted the atomic absorption burner to take up solution at its optimum rate was used while the liquid chromatog. was used at any lower mobile phase flow-rate. Tricyclic antidepressants were determined with a mobile phase of 40% acetonitrile in H₂O at pH 10.7 on uncoated 5- μ m silica particles.

TI Improvements in liquid chromatography column life and method flexibility by saturating the mobile phase with silica

AB Liquid chromatog. column life was prolonged by equilibrating the mobile phase with silica using an appropriate column mounted in the oven ahead of the anal. column. An atomic absorption method was adapted for the detection of Si eluting from the column to measure quant. the loss of silica for different anal. conditions. An injection procedure which permitted the atomic absorption burner to take up solution at its optimum rate was used while the liquid chromatog. was used at any lower mobile phase flow-rate. Tricyclic antidepressants were determined with a mobile phase of 40% acetonitrile in H₂O at pH 10.7 on uncoated 5- μ m silica particles.

IT Silica gel, uses and miscellaneous

RL: USES (Uses)

(in liquid chromatog., mobile phase saturation with)

=> d 2 ibib abs kwic

L3 ANSWER 2 OF 2 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 89:12236 CA

TITLE: Determination of ethinylestradiol in single tablets and its separation from other steroids by high-performance liquid chromatography

AUTHOR(S): Bagon, Kay R.; Hammond, E. W.

CORPORATE SOURCE: Dep. Ind., Lab. Gov. Chem., London, UK

SOURCE: Analyst (Cambridge, United Kingdom) (1978), 103(1223), 156-61
CODEN: ANALAO; ISSN: 0003-2654

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Ethinylestradiol (I) [57-63-6] was rapidly separated from structurally related steroids using a reversed-phase high-performance liquid chromatog. using microfinel silica-gel particles chemical bonded with octadecylsilane. Using this method I was directly assayed down to a limit of 10 μ g in uncoated tablets using a UV detector set at about 212 nm and in sugar-coated tablets after preexn. with Et₂O. The method was sufficiently sensitive to detect I when present with other

steroids as a contaminant.

TI Determination of ethinylestradiol in single tablets and its separation from other steroids by high-performance liquid chromatography

AB Ethinylestradiol (I) [57-63-6] was rapidly separated from structurally related steroids using a reversed-phase high-performance liquid chromatog. using microfinned silica-gel particles chemical bonded with octadecylsilane. Using this method I was directly assayed down to a limit of 10 µg in uncoated tablets using a UV detector set at about 212 nm and in sugar-coated tablets after preextn. with Et2O. The method was sufficiently sensitive to detect I when present with other steroids as a contaminant.

=> s review/ti

L4 59244 REVIEW/TI

=> s uncoated silica gel

15952 UNCOATED

515517 SILICA

488363 GEL

L5 3 UNCOATED SILICA GEL
(UNCOATED(W) SILICA(W) GEL)

=> coated silica gel

COATED IS NOT A RECOGNIZED COMMAND

The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).

=> s coated silica gel

491763 COATED

515517 SILICA

488363 GEL

L6 395 COATED SILICA GEL
(COATED(W) SILICA(W) GEL)

=> s l4 and l5 and l6

L7 0 L4 AND L5 AND L6

=> s separat? epimer?

329427 SEPARAT?

21294 EPIMER?

L8 1 SEPARAT? EPIMER?
(SEPARAT?(W) EPIMER?)

=> d kwic

L8 ANSWER 1 OF 1 CA COPYRIGHT 2007 ACS on STN

IT Epimerization

Resolution (separation)

(epimerization and resolution of naproxen derivs.)

=> s liquid chromatogra? and epimer?

682656 LIQUID

415809 CHROMATOGRAM?

87645 LIQUID CHROMATOGRAM?

(LIQUID(W) CHROMATOGRAM?)

21294 EPIMER?

10/528179

L9 295 LIQUID CHROMATOGRAPHY AND EPIMER?

=> s silica gel and l9

515517 SILICA

488363 GEL

89869 SILICA GEL

(SILICA(W)GEL)

L10 23 SILICA GEL AND L9

=> s uncoated and l10

15952 UNCOATED

L11 0 UNCOATED AND L10

=> d l10 ibib abs 1-23

L10 ANSWER 1 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 143:153556 CA

TITLE: Confirmation of the Structure of (3S)-3-Hydroxyquinine: Synthesis and X-ray Crystal Structure of Its 9-Aceto Analogue

AUTHOR(S): Sarma, P. V. V. Srirama; Han, Dongmei; Deschamps, Jefferey R.; Cook, James M.

CORPORATE SOURCE: Department of Chemistry, University of Wisconsin-Milwaukee, Milwaukee, WI, 53201, USA

SOURCE: Journal of Natural Products (2005), 68(6), 942-944
CODEN: JNPRDF; ISSN: 0163-3864

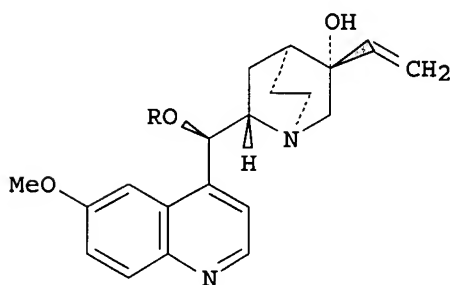
PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 143:153556

GI



AB 3(S)-3-Hydroxyquinine [I; R = H (II)] has been separated from its epimeric mixture at C-3 by conversion into the 9-aceto analog I [R = OAc (III)] followed by chromatog. The mol. structure of III was determined through single-crystal X-ray anal., and this confirms the structure of II, the major metabolite of quinine.

REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 2 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 140:287282 CA

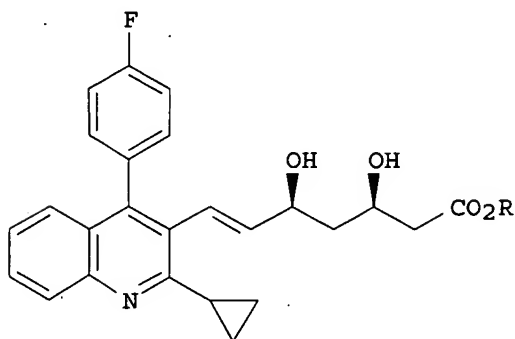
TITLE: Purification of a 3,5-dihydroxy-6-heptenoate isomer

INVENTOR(S): Yoshimura, Yuji; Yasukawa, Masami; Morikiyo, Syuji;

10/528179

PATENT ASSIGNEE(S): Matsumoto, Hiroo; Takada, Yasutaka; Adachi, Michiaki
SOURCE: Nissan Chemical Industries, Ltd., Japan
PCT Int. Appl., 29 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004026838	A1	20040401	WO 2003-JP11643	20030911
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
CA 2499335	A1	20040401	CA 2003-2499335	20030911
AU 2003260963	A1	20040408	AU 2003-260963	20030911
EP 1539698	A1	20050615	EP 2003-797579	20030911
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
CN 1681790	A	20051012	CN 2003-822333	20030911
JP 2006500405	T	20060105	JP 2004-537553	20030911
US 2006167260	A1	20060727	US 2005-528179	20050317
IN 2005KN00588	A	20060303	IN 2005-KN588	20050406
PRIORITY APPLN. INFO.:				
			JP 2002-275015	A 20020920
			WO 2003-JP11643	W 20030911
OTHER SOURCE(S): MARPAT 140:287282				
GI				



I

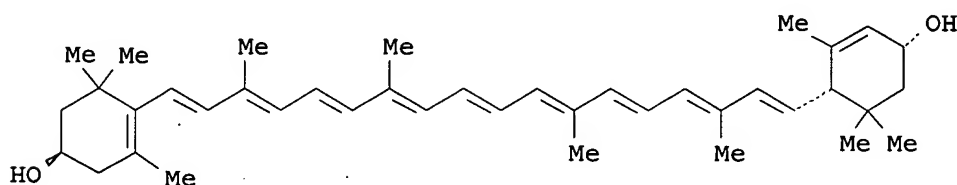
AB An alkyl (3R,5S)-7-[2-cyclopropyl-4-(4-fluorophenyl)quinolin-3-yl]-3,5-dihydroxy-6-heptenoate I [R = alkyl], which is an intermediate for a cholesterol-reducing agent (a HMG-CoA reductase inhibitor), is purified by liquid chromatog. on silica gel.

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 3 OF 23 CA COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 137:93874 CA
 TITLE: Process for obtaining 3'-epilutein via
 epimerization and column chromatography
 INVENTOR(S): Eugster, Conrad Hans; Montoya-Olvera, Ricardo;
 Torres-Quiroga, Jose-Odon
 PATENT ASSIGNEE(S): Industrial Organica, S.A. De C.V., Mex.
 SOURCE: U.S., 7 pp.
 CODEN: USXXAM
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6420614	B1	20020716	US 2000-685321	20001010
WO 2002030769	A9	20021031	WO 2001-MX73	20011010
W: AE, AG, AL, AU, BA, BB, BG, BR, BZ, CA, CN, CO, CR, CU, CZ, DM, DZ, EC, EE, GE, HR, HU, ID, IL, IN, IS, JP, KE, KP, KR, LC, LK, LR, LT, LV, MA, MG, MK, MN, MX, NO, NZ, PL, RO, SG, SI, SK, TT, UA, UZ, VN, YU, ZA				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				

PRIORITY APPLN. INFO.: US 2000-685321 A 20001010
 OTHER SOURCE(S): CASREACT 137:93874
 GI



I

AB A process for obtaining 3'-epilutein (I), by epimerization of a lutein-containing extract with an aqueous solution of a strong organic acid which is slowly added under agitation at room temperature, in the presence of an organic aprotic media, to obtain I (crystals) in a solution which is neutralized with an alkali and extracting the I from said solution by means of an organic media, then washing and drying the crystals and purifying them by chromatog. by means of a chromatog. column. Thus, I was prepared from an enriched lutein (39%) solution via reaction with aqueous 1N. H₂SO₄ in THF for 14 h at room temperature, neutralizing with NH₄OH, partitioning with CH₂Cl₂, then washing the CH₂Cl₂ layer with H₂O and aqueous NaCl, then drying the organic layer over Na₂SO₄; the crystals are purified via chromatog. over silica gel. I can be converted to (3R,3'R)-zeaxanthin via reaction with a strongly alkaline aqueous solution

REFERENCE COUNT: 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 4 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 129:197349 CA
TITLE: High performance liquid chromatography on calixarene-bonded silica gels. II. Separations of regio- and stereoisomers on p-tert-butylcalix[n]arene phases
AUTHOR(S): Gebauer, Sabine; Friebe, Sieglinde; Gubitz, Gerald; Krauss, Gerd-Joachim
CORPORATE SOURCE: Dep. Biochem./Biotechnol., Martin-Luther-Univ. Halle, Halle/S., D-06099, Germany
SOURCE: Journal of Chromatographic Science (1998), 36(8), 383-387
CODEN: JCHSBZ; ISSN: 0021-9665
PUBLISHER: Preston Publications
DOCUMENT TYPE: Journal
LANGUAGE: English
AB The chromatog. behavior of new calix[n]arene-bonded (n = 4, 6, 8) silica gels are described. Cavities of different size and shape are formed depending on the number of aromatic moieties. The differences in ring size were used to study chromatog. selectivities towards analytes of various substance classes, including disubstituted aroms., uracil derivs., and estradiol epimers. The authors' results indicate that these calixarene-bonded phases show a high resolution power for regio- and stereoisomers.

REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 5 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 119:176880 CA
TITLE: Preparation and determination of zinc(II) chlorophylls by reversed-phase high-performance liquid chromatography
AUTHOR(S): Inoue, Hidenari; Imai, Miki; Naemura, Takashi; Furuya, Kenji; Shizuri, Yoshikazu
CORPORATE SOURCE: Faculty of Science and Technology, Keio University, 3-14-1 Hiyoshi, Kohoku-ku, Yokohama, 223, Japan
SOURCE: Journal of Chromatography (1993), 645(2), 259-64
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English
AB A preparative method for zinc(II) chlorophyll a and b [Zn(II)-chl-a and -b] was developed on the basis of their purification by semi-preparative high-performance liquid chromatog. Zn(II)-chl-a and -b and the corresponding epimers were separated and determined with an ODS (C18 chemical bonded silica gel) column using methanol-acetone (75:25, volume/volume) as mobile phase. Linear calibration graphs were obtained over the concentration range 0-50 µg cm⁻³ of each zinc(II) chlorophyll with photometric detection at 425 nm. The present HPLC determination provides an accurate and conventional method with a detection limit of 3.5 ng cm⁻³ for Zn(II)-chl-a, 2.5 ng cm⁻³ for Zn(II)-chl-a' and 3.0 ng cm⁻³ for Zn(II)-chl-b with a relative standard deviation of less than 2.3% (n = 10). The anal. values obtained for synthetic samples by a spectrophotometric method were confirmed to be high compared with those determined by the proposed HPLC method.

L10 ANSWER 6 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 119:117615 CA
TITLE: Method for separation of 24-epimers of
24-hydroxycholesterol derivatives by liquid
chromatography
INVENTOR(S): Saito, Yoichi; Yarino, Tatsuo; Fujii, Takao
PATENT ASSIGNEE(S): Teijin Ltd, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05043591	A	19930223	JP 1991-246480	19910902
JP 2986592	B2	19991206		

PRIORITY APPLN. INFO.: JP 1990-235797 A1 19900907
OTHER SOURCE(S): MARPAT 119:117615

AB An 24-epimeric mixture of 24-hydroxycholesterol derivs. is separated by liquid chromatog. using as a packing material, a silica gel modified with a Si compound R1SiR2R3R4 (R1 = C1-22 aliphatic or C6-10 arom group; R2 - R4 = halo, lower alkoxy, lower alkyl, where at least one of R2 - R4 = halo or lower alkoxy). The packing material shows $K_p = -1$ to 0.2 in an elution liquid system of MeCN/H₂O (30/70), wherein $K_p = [(\text{pyridine retention time}) - (\text{phenol retention time})]/(\text{pyridine retention time})$. Preferably the hydroxycholesterol derivs. separated are $1\alpha, 24$ -dihydroxycholesterol (I) and $1\alpha, 24$ -dihydroxycholesta-5,7-diene (II). Thus, 20% THF solution of a mixture of ($1\alpha, 24R$)- and ($1\alpha, 24S$)-I was fed to a HPLC column YMC-A-802 packed with C4-120-S5, a silica gel modified with BuSiR2R3R4 (at least one of R2 - R4 = Me or Cl), using MeCN/H₂O (6/4) as a mobile phase at room temperature to give ($1\alpha, 24S$)-I with separation degree of 1.2, wherein the separation degree = $[(R\text{-isomer retention time}) - (S\text{-isomer retention time})]/[(R\text{-isomer half width}) + (S\text{ isomer half width})]/(\text{chart speed})$. Addnl. used were YMC-A-202, -302, -402, and YMC-SH-343-50AQ column. II epimers were separated by a column packed with ODS-50AQ.

L10 ANSWER 7 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 117:229369 CA
TITLE: Quantitative derivatization and high-performance
liquid chromatographic analysis of
cyanobacterial heterocyst-type glycolipids
AUTHOR(S): Davey, Mark W.; Lambein, Fernand
CORPORATE SOURCE: Lab. Fysiol. Scheikd., Rijksuniv. Gent, Ghent, B-9000,
Belg.
SOURCE: Analytical Biochemistry (1992), 206(2), 323-7
CODEN: ANBCA2; ISSN: 0003-2697
DOCUMENT TYPE: Journal
LANGUAGE: English

AB Procedures are described for the rapid and quant. anal. of cyanobacterial heterocyst-type glycolipids (HGs) by normal-phase HPLC of their per-O-benzoylated derivs. Total lipids are obtained from 1 mL of nitrogen-fixing cyanobacterial culture by triplicate extraction with chloroform/methanol, 1/1 (volume/volume), and the HGs are isolated from other complex lipids by preparative silica gel TLC. A C18 solid-phase extraction cartridge is used to ensure quant. salt-free recovery of the HGs, and the purified glycolipids are then rendered uv-absorbing by a per-O-benzoylation derivatization reaction for which optimal conditions

have been established. Derivs. are analyzed within 12 min on a 3- μ m silica HPLC column using a linear gradient of 2-propanol in n-hexane and uv monitoring at 230 nm. The reaction product was also used to determine the relative proportions of the glycosyl and galactosyl epimers of individual members of this class of glycolipid.

L10 ANSWER 8 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 116:28266 CA

TITLE: Isolation of doxycycline, 6-epidoxycycline and 2-acetyl-2-decarboxamidometacycline from commercial metacycline by preparative column liquid chromatography on silica gel

AUTHOR(S): Naidong, Weng; Verresen, K.; Busson, R.; Roets, E.; Hoogmartens, J.

CORPORATE SOURCE: Inst. Farm. Wetensch., Kathol. Univ. Leuven, Louvain, B-3000, Belg.

SOURCE: Journal of Chromatography (1991), 586(1), 67-72
CODEN: JOCRAM; ISSN: 0021-9673

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Isolation of doxycycline, 6-epidoxycycline and 2-acetyl-2-decarboxamidometacycline from com. metacycline was achieved by preparative column liquid chromatog. on silica gel, previously impregnated with EDTA. Careful control of the pH of EDTA allowed fine tuning of the separation. The mobile phases were composed of dichloromethane, methanol and 0.1 mM EDTA at pH 9.0 or 6.0. Structures were confirmed with NMR spectroscopy. The presence of doxycycline and its 6-epimer in com. metacycline has not previously been described. The presence of the 2-acetyl derivative was not surprising since analogs 2-acetyl derivs. have been identified in other tetracyclines.

L10 ANSWER 9 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 108:10995 CA

TITLE: Separation of non-polar sesquiterpene olefins from tolu balsam by high-performance liquid chromatography; silver perchlorate impregnation of a prepacked preparative silica gel column

AUTHOR(S): Friedel, Horst Dieter; Matusch, Rudolf

CORPORATE SOURCE: Inst. Pharm. Chem. Lebensmittelchem., Philipps-Univ. Marburg, Marburg, 3550, Fed. Rep. Ger.

SOURCE: Journal of Chromatography (1987), 407, 343-8
CODEN: JOCRAM; ISSN: 0021-9673

DOCUMENT TYPE: Journal

LANGUAGE: English

AB A separation of nonpolar sesquiterpenoid olefins in EtOH exts. of tolu balsam was achieved by using a AgClO₄-loaded HPLC column (LiChrosorb Si 60) and pentane-Et₂CO (80:20 and 99:1) as the mobile phase. The method showed high efficiency and good reproducibility, especially in the separation of epimeric compds. which have similar spectroscopic properties.

L10 ANSWER 10 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 105:205623 CA

TITLE: Separation of allo bile acid stereoisomers by thin-layer and high-performance liquid chromatography

AUTHOR(S): Iida, Takashi; Momose, Toshiaki; Shinohara, Toshiyuki; Goto, Junich; Nambara, Toshio; Chang, Frederic C.

CORPORATE SOURCE: Coll. Eng., Nihon Univ., Koriyama, 963, Japan

SOURCE: Journal of Chromatography (1986), 366, 396-402

11-dehydrocorticosterone, pregnenolone, and testosterone were described and all compds. were separated in a 90% yield. The extrapolation of the conditions used for TLC to preparative HPLC was discussed.

L10 ANSWER 13 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 102:39210 CA

TITLE: Liquid chromatographic separation
of diastereomers and structural isomers on
cyclodextrin-bonded phases

AUTHOR(S): Armstrong, Daniel W.; DeMond, Wade; Alak, Ala; Hinze,
Willie L.; Riehl, Terrence E.; Bui, Khanh H.

CORPORATE SOURCE: Dep. Chem., Texas Tech. Univ., Lubbock, TX, 79409, USA

SOURCE: Analytical Chemistry (1985), 57(1), 234-7

CODEN: ANCHAM; ISSN: 0003-2700

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Compds. (80) were separated from their isomers by liquid chromatog. on cyclodextrin-bonded columns. A variety of structural isomers (including polycyclic aromatic hydrocarbons and prostaglandins), geometric isomers, and steroid epimers were examined. Cyclodextrin-bonded packings appear to be more widely applicable than either normal or reversed-phase packings for these types of sepns. Indeed, compds. that cannot be well resolved on more traditional columns are often easily separated on this stationary phase. The separation mechanism is based on inclusion complex formation and is responsible for the unusual but often predictable selectivities observed

L10 ANSWER 14 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 100:31672 CA

TITLE: Preparation of chlorophylls and pheophytins by
isocratic liquid chromatography

AUTHOR(S): Watanabe, Tadashi; Hongu, Akinori; Honda, Kenichi;
Nakazato, Masataka; Konno, Mitsuo; Saitoh, Sadao

CORPORATE SOURCE: Fac. Eng., Univ. Tokyo, Tokyo, 113, Japan

SOURCE: Analytical Chemistry (1984), 56(2), 251-6

CODEN: ANCHAM; ISSN: 0003-2700

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Isocratic high-performance liquid chromatog. (HPLC) with silica gel as a stationary phase provides a powerful means for rapid preparative isolation (on a 20-50 mg level) of chlorophylls (a, a', b, and b') and pheophytins (a, a', b, and b'). The purity and identical of the isolated pigments were confirmed by complete elemental analyses and anal. HPLC; the purity levels were 99.9, 99.5, 99.5, 99.4, 95, 91, 99.5, and 85% for chlorophyll a, a', b, and b' and pheophytin a, a', b, b', resp., with the sole impurities being almost totally corresponding epimers. UV-visible spectrometric data (in Et₂O, Me₂CO, and C₆H₆) and CD spectra (in C₆H₆) of the purified pigments are presented.

L10 ANSWER 15 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 99:110835 CA

TITLE: Liquid chromatographic assay of
arbaprostil

AUTHOR(S): Peng, G. W.; Sood, V. K.

CORPORATE SOURCE: Pharm. Res. Dev., Upjohn Co., Kalamazoo, MI, 49001,
USA

SOURCE: Journal of Liquid Chromatography (1983), 6(8),
1499-511

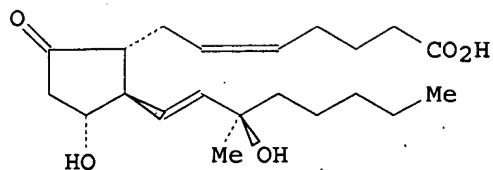
CODEN: JLCHD8; ISSN: 0148-3919

DOCUMENT TYPE: Journal

LANGUAGE: English

10/528179

GI



I

AB Arbaprostil (I) [55028-70-1] was extracted from liquid formulations with Et₂O-CHCl₃, the exts. were evaporated, and I was derivatized with p-nitrophenacyl bromide in the presence of N,N-diisopropylethylamine. The samples were chromatographed on a silica gel column with MeCN-CH₂Cl₂-H₂O (150:350:2.5) mobile phase. The p-nitrophenacyl esters, the 15-S epimer [35700-27-7], and the degradation products of I were separated. When monitored by UV absorption, the degradation products could not be detected as they eluted near the solvent front under the peak of the derivatization reagent. The chromatog. responses were linear with the concns. of I.

L10 ANSWER 16 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 97:203288 CA

TITLE: Determination of 15-methylprostaglandin F_{2α} by derivatization high-pressure liquid chromatography

AUTHOR(S): Wang, Zhongshan; Zhu, Yaohua; Zhang, Shuliang; Zhu, Jiyu; Wang, Meili

CORPORATE SOURCE: Shanghai Inst. Drug Control, Shanghai, Peop. Rep. China

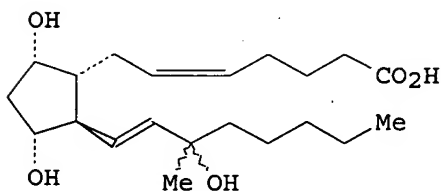
SOURCE: Yaoxue Xuebao (1982), 17(8), 603-8

CODEN: YHHPAL; ISSN: 0513-4870

DOCUMENT TYPE: Journal

LANGUAGE: Chinese

GI



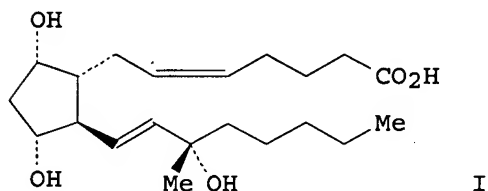
I, ?-OH, ?-Me

II, ?-OH, ?-Me

AB After conversion to their p-bromophenacyl esters at room temperature, epimeric 15-(R)- (I) [35864-81-4] and 15-(S)-methylprostaglandin F_{2α} (II) [35700-23-3] were completely separated by HPLC on a microparticulate silica gel column using CHCl₃-EtOAc (1:1) as the eluent. The derivs. were detected by UV at 257 nm. The ratio of the epimers in a number of samples were accurately determined by this method. Several samples were quant. determined using pure 15-(S)-PGF_{2α} p-bromophenacyl ester (m 85.apprx.86°) as the external standard which was prepared. Other prostaglandins including PGF_{2α}

[551-11-1], PGE1 [745-65-3], PGE2 [363-24-6] and ω -ethyl-13-dehydro-PGF2 α [36950-85-3] were also determined by the same method and their relative retention time compared to 15-(S)-PGF2 α p-bromophenacyl ester were given. The method proposed was suitable for accurate determination of I and II and their relative contents in samples.

L10 ANSWER 17 OF 23 CA COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 94:109418 CA
 TITLE: Comparison of two high-pressure liquid chromatographic assays for carboprost, a synthetic prostaglandin
 AUTHOR(S): Brown, Leo W.; Carpenter, Bruce E.
 CORPORATE SOURCE: Control Res. Dev., Upjohn Co., Kalamazoo, MI, 49002, USA
 SOURCE: Journal of Pharmaceutical Sciences (1980), 69(12), 1396-9
 CODEN: JPMSAE; ISSN: 0022-3549
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 GI

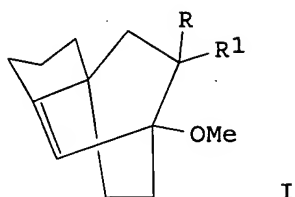


AB A high-pressure liquid chromatog. assay for carboprost tromethamine (I tromethamine) [58551-69-2] as the bulk drug and in a sterile solution formulation is described. The procedure involves derivatization of the prostaglandin to form the UV-absorbing naphthacyl ester, which then is chromatographed on a silica gel column using CH2Cl2-1,3-butanediol-H2O (496:4:0.25) as the mobile phase. This procedure is compared with a nonderivatization procedure with refractive index detection. Both procedures sep. the 15R-epimer of I [35864-81-4] from I, but only the derivatization procedure seps. the 5-trans-isomer of I [76498-29-8]. Possible reasons for the better separation using the derivatization procedure are discussed. Both procedures gave a coefficient of variation of .apprx.1% for I. The derivatization procedure gave a coefficient of variation of .apprx.7% for the 15R-epimer and 5-trans-isomer when present at 2% of the I level.

L10 ANSWER 18 OF 23 CA COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 93:185599 CA
 TITLE: Liquid chromatographic resolution of epimeric Diels-Alder adducts on silica gel using binary solvent systems
 AUTHOR(S): Hara, Shoji; Ohkuma, Toshikazu; Nagaoka, Hiroto; Yamada, Yasuji
 CORPORATE SOURCE: Tokyo Coll. Pharm., Tokyo, 192-03, Japan
 SOURCE: HRC & CC, Journal of High Resolution Chromatography and Chromatography Communications (1980), 3(4), 193-4
 CODEN: HJCJDB; ISSN: 0344-7138
 DOCUMENT TYPE: Journal

10/528179

LANGUAGE: English
GI



AB Resolution of I (R = Cl R1 = CN; R = CN, R1 = Cl) was accomplished on a silica gel column using a hexane-2-propanol system.

L10 ANSWER 19 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 89:175853 CA

TITLE: Separation of peracetylated mono- and disaccharides and quantitative analysis of guaran by high-performance liquid chromatography on silica gel

AUTHOR(S): Thiem, Joachim; Schwentner, Jens; Karl, Horst; Sievers, Axel; Reimer, Joachim

CORPORATE SOURCE: Inst. Org. Chem. Biochem., Univ. Hamburg, Hamburg, Fed. Rep. Ger.

SOURCE: Journal of Chromatography (1978), 155(1), 107-18
CODEN: JOCRAM; ISSN: 0021-9673

DOCUMENT TYPE: Journal

LANGUAGE: English

AB High-performance liquid chromatog. on silica gel was successfully applied to the separation of 15 monosaccharide peracetates. The solvent system Me₂CO-n-hexane gave the most efficient sepns. at the optimal flow-rate. Dependences of retention times or capacity factors on flow-rate or pressure were determined for all derivs. The chromatograms obtained showed clear sepns. of all derivs., including epimers and anomers. Similarly, 12 disaccharide peracetates were analyzed. The best solvent system was Me₂CO-pentane, and again the resulting chromatograms showed excellent sepns. For detection both a UV photometer and a refractive index detector could be used, depending on the eluent. Acid cleavage and subsequent peracetylation of the polysaccharide guaran gave a mixture of peracetylated monosaccharides. The interpretation of the chromatogram led to qual. results and, after careful calibration, to quant. results for the ratio of galactose to mannose residues in guaran.

L10 ANSWER 20 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 66:28981 CA

TITLE: Dehydrochlorination of 3 β -chloro-5 β -cholestane. New syntheses of 5 β -cholest-2-ene and 5 β -cholest-3-ene

AUTHOR(S): Bellucci, Giuseppe; Macchia, F.; Malaguzzi, Valerio

CORPORATE SOURCE: Univ. Pisa, Pisa, Italy

SOURCE: Tetrahedron, Supplement (1966), (41), 4973-8
CODEN: TETSAE; ISSN: 0563-2072

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Elimination reactions in the 3-substituted-5 β -cholestanes (I) were investigated by means of gas liquid chromatography at 190° over 2 m. glass columns containing 1% NGS on Chromosorb W.

Quinoline dehydrochlorination of I (R = Cl) (II), m. 122-4°, yielded 5 β -cholest-3-ene (III) and 5 β -cholest-2-ene (IV) in 55:45 ratio, showing retention times of 1.06, 0.94 relative to 5 α -cholestane. Chromatography of the mixture over silica gel-AgNO₃ and elution with petr. ether gave pure III. Reduction of 4 β -bromo-5 β -cholestan-3-one, m. 110-12°, with NaBH₄ in absolute alc. at 25° gave a mixture of epimeric bromohydrins, transformed by refluxing with Zn in AcOH to pure III, m. 48-9° (EtOAc-MeOH), $[\alpha]$ 23 D 19.2° (c 1.583, CHCl₃); 3 α , 4 β -dibromo derivative m. 98.5-100.0°. Abnormal opening of 4 β , 5-epoxy-5 β -cholestan-3-one with H₂SO₄-Me₂CO followed by acetylation and the resulting 2 α -acetoxycholest-4-en-3-one hydrogenated over 10% Pd-C in MeOH-dioxane saturated with NaHCO₃ gave 65% 2 α -acetoxy-5 β -cholestan-3-one (V), m. 137-9° (petr. ether), $[\alpha]$ 28 D 0.5°, readily epimerized in AcOH-HBr to the more stable 2 β -acetoxy-5 β -cholestan-3-one, m. 150.5-2.0° (petr. ether), $[\alpha]$ 27 D -4°. V submitted to the Wolff-Kishner reaction under Huang-Minlon conditions yielded IV together with about 30% 5 β -cholestane (VI), m. 70-1°. The mixture brominated at 18° in CHCl₃ and heated (N atmospheric) at 180-200° to isomerize the dibromide of II to a more stable form and the product chromatographed on silica gel and eluted with petr. ether gave in succession VI and a dibromide, m. 165-7° (Me₂CO), $[\alpha]$ 27 D 71.4° (c 1.060, CHCl₃), refluxed in AcOH with Zn to yield pure IV, m. 47.5-8.0° (Me₂CO), $[\alpha]$ 28 D 19.9° (c 1.720, CHCl₃). IV brominated as above gave a dibromide, m. 74-6° (Me₂CO), isomerized to the above-mentioned compound. It was surmised that the compds., m. 74-6° and 165-7°, are the diaxial and diequatorial 2 α , 3 β - and 2 β , 3 α -dibromo-5 β -cholestanes.

L10 ANSWER 21 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 64:104458 CA
 ORIGINAL REFERENCE NO.: 64:19688f-h,19689a-c
 TITLE: Terpenoids. LXXXVII. Structure of nardol
 AUTHOR(S): Sastry, S. D.; Maheshwari, M.; Bhattacharyya, S. C.
 CORPORATE SOURCE: Natl. Chem. Lab., Poona, India
 SOURCE: Tetrahedron Letters (1966), (10), 1035-42
 CODEN: TELEAY; ISSN: 0040-4039
 DOCUMENT TYPE: Journal
 LANGUAGE: English

AB cf. CA 62, 4058f; preceding abstract Isolation from the dark brown variety of Nardostachys jatamansi roots by use of a low temperature solvent extraction procedure gave 4.38% yield of a concentrate sapd. to yield 2.2:97.8 acidic-neutral fractions. The high vacuum distillate from the neutral fraction chromatographed over Al₂O₃ (activity III) and elution with petr. ether, C₆H₆, Et₂O, and alc. gave 4.5% calarene, 4.5% aristolene, 0.48% valeranone, and several unidentified compds. from the petr. ether fraction. Careful rechromatography of the C₆H₆ eluate on Al₂O₃ (activity II) yielded 0.36% of a new alc., nardol (I), C₁₅H₂₆O, b_{0.5} 120-5°, n_{26D} 1.5005, $[\alpha]$ 27D -10.17° (c 2.36, CHCl₃), ν 1648, 895 cm.⁻¹, λ 220 m μ (ϵ 560), giving a pos. C(NO₂)₄ yellow color but containing only 1 double bond (quant. hydrogenation over PtO₂ in alc. AcOH). I was stable in Jones reagent and contains a tertiary OH group. I dehydrogenated 13 hrs. with Se yielded Se-guaiazulene (trinitrobenzene adduct m. 148-9°). S dehydrogenation gave S-guaiazulene (trinitrobenzene adduct m. 147-8°). Continued dehydrogenation (48 hrs.) gave a mixture of naphthalenic hydrocarbons, eudalene and cadulene (25% 40:60), showing that I contains the basic azulenic skeleton. The N.M.R. spectrum of I showed a methylenic doublet

at τ 5.45 and the presence of Me₂CH and Me groups at τ 9.15, 9.18, and 8.89. The position of the double bond was demonstrated by ozonization to an oxo alc. (II), ν 1712 cm.⁻¹ with a CO group on a 7-membered ring. The N.M.R. spectrum of dihydronardol (III), τ 8.6, 8.95, 9.06, 9.15, 9.17 showed the presence of 1 Me group on a C atom carrying an O function and 3 secondary Me groups. On the basis of the above data structures were assigned to I, II, and III, resp. The position of the OH group at C was confirmed. III benzoate was pyrolyzed and the epimeric hydrocarbon mix., ν 803, 892, 1650 cm.⁻¹ separated by preparative thin layer chromatography on silica gel impregnated with AgNO₃ to give a hydrocarbon, N.M.R. γ 4.9, 8.39 [(J = 2 cycles/sec. (cps.)), 9.05, 9.11, 9.14, and its isomer, τ 4.67, 8.19 (J = 2 cps.), 9.13 (J = 6 cps.), indicating that the mixts. may be represented by the given structures (IV or V). Dehydration of the mixture of C₁₀ epimers, III with SOCl₂ in C₅H₅N also gave the epimeric mixture IV, V and the mode of dehydration suggested the axial nature of the OH group. The epimeric mixture of IV, V hydrogenated catalytically gave a mixture of saturated hydrocarbons, $[\alpha]_D^{27}$ -31.06°, shown by gas liquid chromatographic analysis to be a mixture of 3 epimers in the ratio 65:25:10. The retention time of the major constituent was identical with that of guaiane, though the possibility of trans ring junctures in the major constituent cannot be ruled out.

L10 ANSWER 22 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 63:45835 CA

ORIGINAL REFERENCE NO.: 63:8221b-h,8222a

TITLE: Macrocyclic musk compounds. IX. New synthesis of cyclohexadecenone and cyclohexadecanone from aleuritic acid

AUTHOR(S): Mathur, H. H.; Bhattacharyya, S. C.

CORPORATE SOURCE: Natl. Chem. Lab., Poona, India

SOURCE: Tetrahedron (1965), 21(6), 1537-40

CODEN: TETRAB; ISSN: 0040-4020

DOCUMENT TYPE: Journal

LANGUAGE: English

GI For diagram(s), see printed CA Issue.

AB cf. CA 58, 6709f; 63, 4177f. Aleuritic acid (100 g.) was stirred in pure dry Me₂CO containing 2 ml. concentrated H₂SO₄ and the isopropylidene derivative oxidized

with 75 g. KMnO₄ (CA 60, 11839h), filtered, and the MnO₂ sludge dispersed in 500 ml. H₂O and bubbled through with SO₂, the oily product deacetylated by boiling with stirring in 500 ml. H₂O containing 25 ml. 5N H₂SO₄ 30 min., the cooled mixture filtered, and the washed and dried product crystallized from alc. gave 68 g. HO₂C(CH₂)₅CH(OH)CH(OH)(CH₂)₇CO₂H (I), m. 129.5-30°, ν 3100, 1680, 1130 cm.⁻¹ I (100 g.) treated with 1.36 1. 15% HBr in AcOH, the crude dibromo derivative (140 g.) esterified, and the diethyl ester (n_D²⁰ 1.4865) debrominated with Zn dust in alc. gave 86 g. trans-EtO₂C(CH₂)₆CH:CH(CH₂)₇-CO₂Et (II), b_{0.05} 145-6°, n_D²⁰ 1.4510, ν 969 cm.⁻¹ (trans-CH:CH), converted to trans-hexadec-7-enedioic acid, m. 96-7° (AcOH), ν 2860, 1684, 962 cm.⁻¹ II (20 g.) cyclized with Na in xylene gave 14 g. mixture of acyloins, b_{0.05} 133-8°, ν 3300, 1711, 966 cm.⁻¹. The acyloins were acetylated and the mixed acetates (11.6 g., b_{0.04} 131-3°, ν 1732, 1242, 970 cm.⁻¹) reduced with 6 g. Ca in 750 ml. liquid NH₃, the product (9.5 g., n_D²⁰ 1.4980) chromatographed over 190 g. Al₂O₃ (grade II) and eluted with 100 ml. C₆H₁₄ gave a ketone-free fraction (0.5 g.), rechromatographed over 50 g. Al₂O₃ (grade I), eluted with C₆H₁₄, and the eluate distilled over 0.37 g. Na to give material, b_{1.0} 180°, n_D²⁰ 1.4695, ν 2920, 2646, 1439 1361, 1333, 1290, 1147, 1012, 962, 883, 716 cm.⁻¹, containing some impurity

according to gas-liquid chromatographic analysis. The NMR signal at τ 9.13 indicated the presence of a secondary Me group possibly formed due to ring contraction. Elution with 400 ml. C₆H₁₄ gave 0.46 g. material, distilled to yield the 2 cyclohexadecenones (III, 0.37 g.), C₁₆H₂₈, b.p. 170°, n_D 1.4967, ν 2950, 2874, 1709, 1458, 1402, 1372, 1290, 1029, 971 cm.⁻¹; semicarbazone m. 180-1°, ν 3445, 3160, 1692, 1581, 1311, 1294, 1208, 1137, 1104, 1079, 965, 764, 722 cm.⁻¹ Further elution with 100 ml. 1:2 C₆H₁₄-C₆H₆ gave an unidentified fraction (0.79 g.), n_D 1.5041, ν 3505, 1708 cm.⁻¹ Elution with 600 ml. C₆H₆ yielded 1.93 g. material, n_D 1.5005, ν 3355, 1703 cm.⁻¹, rechromatographed over 100-fold amount Al₂O₃ and eluted with 1:1 C₆H₁₄-C₆H₆, to give a sticky solid, m. 61-70°, repeatedly sublimed to yield the alcs. (IV), m. 70-2°, ν 3320, 1455, 1347, 1229, 1189, 1077, 1051, 1009, 964, 869, 722 cm.⁻¹ Further elution with 500 ml. Et₂O and the fraction (1.86 g.) crystallized from C₆H₁₄ yielded trans-cyclohexadec-8-ene-1,2-diol (V), m. 87.0-8.5°, ν 3355, 2355, 1292, 1174, 1133, 1072, 1012, 964 cm.⁻¹ Elution with 500 ml. CHCl₃ gave an epimer of V, m. 76-7° (C₆H₁₄), ν 3250, 963 cm.⁻¹, whereas the final elution with 350 ml. alc. yielded the other epimer, m. 111-12° (C₆H₁₄), ν 3200, 964 cm.⁻¹ II hydrogenated over Raney Ni gave 18.6 g. saturated ester EtO₂C(CH₂)₁₄CO₂Et, m. 37.0-7.5°, cyclized with Na in xylene to give 9.75 g. acyloin, b.p. 139-41°, ν 3400, 1716, 1453, 1410, 1353, 1297, 1060 cm.⁻¹; acetate, b.p. 125-7°, ν 1729, 1440, 1360, 1237, 1114, 1022 cm.⁻¹ The acetate (7.1 g.) reduced with 3.85 g. Ca in 500 ml. liquid NH₃ and the solid product (5.64 g.) chromatographed on 100 g. Al₂O₃ (grade II), eluted with 50 ml. C₆H₁₄, and the hydrocarbon, 967 cm.⁻¹, treated with oleum, chromatographed over 60-fold amount of activated silica gel, and eluted with C₆H₁₄, the eluate (0.45 g.) rechromatographed over 60 g. Al₂O₃ and eluted with C₆H₁₄ gave 0.36 g. hydrocarbon, distilled over 0.18 g. Na to yield 87% pure C₁₆H₃₂, b.p. 150°, n_D 1.4600, ν 2830, 1458, 1375, 1300, 721 cm.⁻¹, N.M.R. signal at τ 9.12, indicating the presence of a related hydrocarbon containing a secondary Me group. Elution with 450 ml. C₆H₁₄ gave 0.43 g. fraction, distilled to yield material, b.p. 158°, λ 234 m μ (ϵ 3709), apparently containing some α,β -unsatd. ketone. The material hydrogenated and the product crystallized from MeOH gave cyclohexadecanone, m. 62.0-3.5°, λ 1711, 1412, 1284, 1206, 1177, 1149, 1123, 1086, 1046, 730 cm.⁻¹; semicarbazone m. 184.0-4.5°, ν 3445, 3085, 2340, 1660, 1581, 1345, 1231, 1080, 770 cm.⁻¹ Further elution with 900 ml. 1:1 C₆H₁₄-C₆H₆ gave 0.61 g. fraction, crystallized from MeOH and sublimed to yield pure cyclohexadecanol, m. 82.0-3.5°, ν 3310, 2940, 1335, 1274 cm.⁻¹ Elution with 500 ml. Et₂O and 200 ml. alc. produced 0.83 g. and 2.14 g. fractions, repeatedly crystallized from C₆H₁₄ to give the epimeric cyclohexadecane-1,2-diols, m. 102-3°, and 107-8°, ν 3240, 2350, 1299, 1171, 1149, 1129, 1058, 917 cm.⁻¹ Two intermediate fractions which absorbed in both the hydroxyl and carbonyl regions were not processed further.

L10 ANSWER 23 OF 23 CA COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 58:40176 CA
 ORIGINAL REFERENCE NO.: 58:6870g-h,6871a-h
 TITLE: Stereochemistry of occidentalol and its hydrogenation products
 AUTHOR(S): v. Rudloff, E.; Erdtman, H.
 CORPORATE SOURCE: Natl. Regional Res. Lab., Saskatoon, Can.
 SOURCE: Tetrahedron (1962), 18, 1315-29
 CODEN: TETRAB; ISSN: 0040-4020
 DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB cf. CA 55, 23938e. The milled heartwood of a mature Eastern white cedar (*Thuja occidentalis*) extracted with Me₂CO and the dark viscous product poured into Et₂O, the decanted Et₂O extracted 3 times with 10% KOH and the washed and dried extract evaporated, the neutral residue fractionally distilled through a spinning band column at 18 mm. and the fraction, b₇₈ 148-9°, purified by crystallization from petr. ether and by sublimation gave occidentol

(I), m. 97.5-8.0°, [α]_{25D} 363.2° (c 1.6, CHCl₃). I was analyzed by gas-liquid chromatography by using coiled C7 tubing (6 ft. + 0.25 in.) containing adipate polyethylene glycol polyester, polyphenyl ether, and SE-30 silicone polymer on Chromosorb W in the ratio of 1:6 and was better than 99-5% pure. The relative retention time (RRT) with respect to cedrol on adipate polyester at 160° in 60 ml. He/min. was 0.92. I (0.5 g.) in CH₂Cl₂ at -70° ozonized and the blue solution extracted with H₂O, the extract treated with excess PhNHNH₂.HCl and the phenylhydrazones chromatographed from C₆H₆ on silica gel, eluted with C₆H₆ and the fraction crystallized from C₆H₆-petr. ether gave glyoxal bis(phenylhydrazone), m. 173-5°. I (5.0 g.) in 20 ml. alc. hydrogenated with 0.5 g. prereduced Pd-C and the filtered solution diluted with 5 ml. H₂O, kept at 2-5° and the precipitate, m. 81-6°, recrystd., sublimed and recrystd. gave dihydrooccidentol (II), m. 86.0-7.8°, [α]_{25D} 65.5° (c 2.3, CHCl₃), RRT 1.06, containing less than 2-3% each of tetrahydrooccidentol (III), RRT 1.15, and epitetrahydrooccidentol (IV), RRT 0.90. Mixed m.p. of II with eudesmol (V), RRT 1.46 was 72-84°, showing their apparent nonidentity. II (1.0 g.) in 8 ml. MeOH and 2 ml. AcOH hydrogenated slowly with 0.1 g. prereduced PtO₂, the filtered solution poured slowly into saturated aqueous NaHCO₃ and extracted

with Et₂O

gave material showing 2 peaks of RRT 1.15 (91%) and 0.90 (9%); crystallized from petr. ether, sublimed and recrystd. to yield practically pure III, m. 82.54.0°, [α]_{25D} 42.5° (c 3.4, CHCl₃), mixed m.p. with dihydroeudesmol (VI, m. 85.5-6.0°, [α]_{25D} 18.1°, RRT 1.24) 60-82°. I (3.0 g.) in 90% aqueous alc. hydrogenated with 0.3 g. Pd-C and the filtered solution diluted with H₂O, extracted with Et₂O and the product taken up in 50 ml. Me₃COH, stirred intermittently with portionwise alternate addition of 10 g. KMnO₄ and 0.5 g. KOH in 200 ml. H₂O and the mixture stirred 3 hrs., kept 16 hrs. at 20° and excess KMnO₄ destroyed by addition of Na₂S₂O₆, filtered and the filtrate and 90% aqueous Me₃COH washings evaporated to a small volume, the crystalline product (0.3 g.) recrystd. from

aqueous

MeOH and the material, m. 114.5-16.0°, sublimed and recrystd. gave IV, m. 116-17°, [α]_{25D} 9.3° (c 1.6, CHCl₃), mixed m.p. 73-88°, with VI. Hydrogenation of I in dry C₅H₆ gave only 3-5% IV suggesting that 1,4 addition may be involved and that IV is the C-4 epimer of III. If the HOCMe₂ group in I and its hydrogenation products is equatorial, then these compds. can differ only from V and VI at the ring juncture and therefore I must have cis-fused rings. Com. V fractionally distilled, the fraction, b₁₀ 140-2°, recrystd. repeatedly from petr. ether or MeOH, sublimed, and recrystd. gave a pure isomeric mixture of α - and β -V, m. 81-2°, [α]_D 35.0° (c 2.2, CHCl₃). V (5.0 g.) in 20 ml. MeOH hydrogenated with 0.25 g. prereduced PtO₂, the filtered solution diluted with 3-5 ml. H₂O, the solution chilled to 2-5°, and the product recrystd. and sublimed gave VI, m. 85.5-6.0°, [α]_{25D} 18.1° (c 2.16, CHCl₃). V (2.0 g.) in 16 ml. MeOH and 4 ml. AcOH hydrogenated 5-6 hrs. at 45-55° with 0.2 g. Pd-C and the filtered diluted solution chilled gave a mixture showing 2 peaks with RRT 0.98 and 1.27 in 53:47 ratio on gas-liquid

chromatography, separated to give epidihydroeudesmol (VII), m. 75.0-82.5° (purified by sublimation and recrystn. to constant m. 73.0-5.5°, $[\alpha]_D$ 12.8° (c 1.9, CHCl₃), RRT 0.96, and VI, m. 85.0-7.5°, $[\alpha]_D$ 15°. Treatment of VI and VII with H and Pd-C 24 hrs. at 20° gave no evidence of epimerization. Assuming equatorial orientation of the HOCMe₂ group, VII must be the C-4 epimer and III and IV are the C-4 epimers of VIII. To eliminate the possibility that I and its derivs. are merely C-7 α -epimers of V, the HOCMe₂ group was removed by oxidation with CrO₃. VI (1.4 g.) in 8 ml. AcOH containing a few drops of H₂SO₄ and stirred at 80° on a steam bath 1 hr. with dropwise addition of 1.25 g. CrO₃ in 24 ml. 1:2 H₂O-AcOH, the solution kept 2 hrs. at 80-5° and treated with 1 ml. MeOH, the cooled CrO₃-free solution neutralized with NaHCO₃ and extracted with Et₂O, the product analyzed

by

gas-liquid chromatography and fractionated on silica gel, eluted with petr. ether to give 0.05 g. hydrocarbon and 0.30 g. 5,9-dimethyl-3-decalone (IX) (semicarbazone m. 219-21°) and finally eluted with Et₂O or CHCl₃ gave 0.35 g. unreacted VI. III and IV similarly degraded and analyzed gave liquid ketone fractions and starting materials, but the ketone fractions did not show a peak at RRT 0.90, nor was the crystalline semicarbazone, m. 222° obtained. Accordingly since III and IV failed to give IX they must differ from VI at the ring juncture as assumed.

=> FIL STNGUIDE

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

110.11

110.32

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE

TOTAL

ENTRY

SESSION

CA SUBSCRIBER PRICE

-18.25

-18.25

FILE 'STNGUIDE' ENTERED AT 10:29:18 ON 01 FEB 2007

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FILE CONTAINS CURRENT INFORMATION.

LAST RELOADED: Jan 26, 2007 (20070126/UP).

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YOU HAVE REQUESTED DATA FROM FILE 'CA' - CONTINUE? (Y)/N:Y

L10 ANSWER 9 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

108:10995 CA

TITLE:

Separation of non-polar sesquiterpene olefins from tolu balsam by high-performance liquid chromatography; silver perchlorate impregnation of a prepacked preparative silica gel column

AUTHOR(S):

Friedel, Horst Dieter; Matusch, Rudolf

CORPORATE SOURCE:

Inst. Pharm. Chem. Lebensmittelchem., Philipps-Univ. Marburg, Marburg, 3550, Fed. Rep. Ger.

SOURCE:

Journal of Chromatography (1987), 407, 343-8
CODEN: JOCRAM; ISSN: 0021-9673

10/528179

DOCUMENT TYPE: Journal
LANGUAGE: English

- AB A separation of nonpolar sesquiterpenoid olefins in EtOH exts. of tolu balsam was achieved by using a AgClO₄-loaded HPLC column (LiChrosorb Si 60) and pentane-Et₂CO (80:20 and 99:1) as the mobile phase. The method showed high efficiency and good reproducibility, especially in the separation of epimeric compds. which have similar spectroscopic properties.
- TI Separation of non-polar sesquiterpene olefins from tolu balsam by high-performance liquid chromatography; silver perchlorate impregnation of a prepacked preparative silica gel column
- AB A separation of nonpolar sesquiterpenoid olefins in EtOH exts. of tolu balsam was achieved by using a AgClO₄-loaded HPLC column (LiChrosorb Si 60) and pentane-Et₂CO (80:20 and 99:1) as the mobile phase. The method showed high efficiency and good reproducibility, especially in the separation of epimeric compds. which have similar spectroscopic properties.
- IT Sesquiterpenes and Sesquiterpenoids
RL: BIOL (Biological study)
(olefins, separation from tolu balsam by HPLC of, silver-impregnated silica gel column in)
- IT Alkenes, analysis
RL: ANST (Analytical study)
(sesquiterpenoid, separation from tolu balsam by HPLC of, silver-impregnated silica gel column in)
- IT Chromatography, column and liquid
(high-performance, stationary phases, silver-impregnated silica gel, for sesquiterpenoid olefins separation in tolu balsam)
- IT Balsams
(tolu, sesquiterpenoid olefins separation from, by HPLC, silver-impregnated silica gel column in)
- IT 489-39-4 489-40-7 2387-78-2 3856-25-5 22567-17-5 25246-27-9
111778-06-4 111820-84-9 111821-79-5 111900-50-6 111900-51-7
RL: BIOL (Biological study)
(separation of, from tolu balsam by HPLC, silver-impregnated silica gel column in)
- IT 7783-93-9, Silver perchlorate
RL: BIOL (Biological study)
(silica gel column impregnated with, for HPLC separation of non-polar sesquiterpenoid olefins)

=> d his

(FILE 'HOME' ENTERED AT 10:23:38 ON 01 FEB 2007)

FILE 'CA' ENTERED AT 10:23:47 ON 01 FEB 2007

L1 89869 S SILICA GEL
L2 4609 S L1 AND LIQUID CHROMATOGRAPH?
L3 2 S UNCOATED AND L2
L4 59244 S REVIEW/TI
L5 3 S UNCOATED SILICA GEL
L6 395 S COATED SILICA GEL
L7 0 S L4 AND L5 AND L6
L8 1 S SEPARAT? EPIMER?
L9 295 S LIQUID CHROMATOGRA? AND EPIMER?
L10 23 S SILICA GEL AND L9
L11 0 S UNCOATED AND L10

FILE 'STNGUIDE' ENTERED AT 10:29:18 ON 01 FEB 2007

10/528179

FILE 'CA' ENTERED AT 10:30:41 ON 01 FEB 2007

FILE 'STNGUIDE' ENTERED AT 10:30:41 ON 01 FEB 2007

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---Logging off of STN---

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Executing the logoff script...

=> LOG Y

COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	0.06	113.96
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	0.00	-18.98

STN INTERNATIONAL LOGOFF AT 10:31:14 ON 01 FEB 2007

CODEN: JOCRAM; ISSN: 0021-9673

DOCUMENT TYPE: Journal
LANGUAGE: English

AB Twenty-six allo bile acids were separated by normal-phase TLC (NP-TLC) and reversed-phase TLC (RP-TLC) as their Me esters; they were also separated by reversed-phase HPLC as their 4-nitrophthalimidemethyl esters. NP-TLC and RP-TLC were on silica gel 60F254 and octadecyl-bonded silica gel RP-18 F2545, resp.; HPLC was on Nova-Pak C18 RP. Results were compared to those for the corresponding 5 β epimers.

L10 ANSWER 11 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 105:178553 CA

TITLE: Reversed phase high performance liquid chromatographic separation of tetracycline, anhydrotetracycline and their C4-epimers

AUTHOR(S): Iwuagwu, M. A.

CORPORATE SOURCE: Dep. Pharm. Pharm. Technol., Univ. Benin, Benin City, Nigeria

SOURCE: Nigerian Journal of Pharmaceutical Sciences (1986), 2(1), 83-90

CODEN: NJPSEZ; ISSN: 0189-322X

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Tetracycline (I) [60-54-8] and its impurities, epitetracycline [79-85-6], anhydrotetracycline [1665-56-1], and epianhydrotetracycline [7518-17-4] were separated and determined in pharmaceuticals by HPLC. ODS columns

bonded to glass beads or silica gel and a mobile phase consisting of MeCN, MeOH and 0.2M phosphate buffer in different ratios were used. The chromatog. was carried out at room temperature, at pH 2.5 with flow rate of 1.5 mL \cdot min⁻¹. The method is simple, rapid, sensitive and suitable as a stability-indicating assay method for study of the kinetics of I degradation. The recovery of I was 99.8% with relative standard deviations for I, epitetracycline, anhydrotetracycline and epianhydrotetracycline of 0.11, 0.78, 0.40 and 0.96%, resp.

L10 ANSWER 12 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 105:746 CA

TITLE: Separation of 7 α - and 7 β -methoxycarbonylmethyl steroids by preparative high-performance liquid chromatography: comparison with thin-layer chromatography

AUTHOR(S): Charpentier, Bruno; Dingas, Alexandre; Duval, Daniele; Emiliozzi, Romeo

CORPORATE SOURCE: Lab. Chim. Phys. Org., UER Domaine Mediterr., Nice, 06034, Fr.

SOURCE: Journal of Chromatography (1986), 355(2), 427-33

CODEN: JOCRAM; ISSN: 0021-9673

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The 7 α - and 7 β -carboxymethyl steroid derivs. have been used as heptens in RIA or as ligands in affinity chromatog. TLC separation was performed on activated (110 $^{\circ}$) silica gel plates with solvents of hexane-EtOAc (30:70), EtOAc, and benzene-(Et)₂O (60:40, 30:70) with UV detection. HPLC was performed on μ Porasil or reversed-phase μ Bondapak columns with a mobile phase mixture of hexane-EtOAc-CH₂Cl₂ of various combinations. Conditions for the separation of each pair of epimers derived from androsterone, cortisone,